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effect of radiation energy on flexible containers

Items made of paper, plastics, textiles, and wood, when exposed to normal environmental stresses of heat, light, air (oxygen, carbon dioxide, moisture vapor, sulfur dioxide, and other gases) gradually lose their useful properties through oxidation and other deterioration. This deterioration will begin and continue at a rate which depends upon the nature of the substance and the severity of the environmental conditions.

When polymeric substances are exposed to gamma or beta radiation ions, free radicals form in the solid material resulting in cross-linking or chain scission. Simultaneously, unsaturated bonds, hydrogen, and other gases may be formed and, where the irradiation is conducted in air, oxidation may also occur. The net result is governed by the predominant reaction, which in turn depends upon the chemical structure of the polymer. Ultraviolet radiation and, to a lesser degree, infrared radiation will also cause the formation of ions and free radicals in polymeric substances and promote oxidation. The principal difference in the main course of change depends upon the quantity of energy transmitted to the polymeric molecule, the rate of such energy transfer, and the threshold energy (minimum energy required to initiate a given reaction) of the various possible reactions. With heat or small quantities of infrared radiation the energy imparted is small, and generally only reactions with very low threshold values will occur. With increasing energy content of the incident radiation, chemical changes with higher threshold values can be induced along with changes that

would normally be induced by low energy radiations.

A considerable number of investigations have been conducted with polymeric substances exposed to heat, infrared radiation, ultraviolet radiation, beta radiation, gamma radiation, and to combinations of these various types of "energies." When polystyrene is exposed to heat and ultraviolet radiation, hydrogen, water vapor, and carbon dioxide are formed along with numerous hydrocarbons and carbonyl type compounds *(1, 49)*. In addition, the solubility of the polymer is decreased by crosslinking. Similar changes have been observed when polystyrene has been exposed to gamma, beta, or pile irradiation. At present it is difficult to correlate data since quantitative absorption studies have not been made with infrared and ultraviolet radiation.

The changes induced by radiation have a profound effect on polymeric materials. With crosslinking there is a marked increase in molecular weight and a conversion of the molecule from a linear planar structure to a three-dimensional configuration. Principally, this results in an increase of tensile strength, flexural strength, and softening-point elevation, and there is a corresponding decrease in elongation, crystallinity, and solubility. Since the properties of polymers are influenced by secondary forces of molecular attraction, a shortening of the polymeric chain, through scission, will result in a loss of properties associated with the polymer such as tensile strength and flexural strength. The formation of unsaturated, conjugated, polyene bonds affects the color, but the effect on physical properties is small. The most serious aspect of unsaturated bonds is their susceptibility to oxidation and chain scission. The formation of gases within the plastic will result in increased porosity or foaming either during the radiation process or on subsequent exposure to heat.

The relative stability of various chemical groupings is shown in table 1.

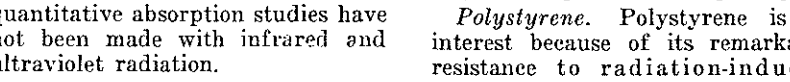
relative stability of flexible materials

The more common food packaging materials have been studied in varying degrees; the results of these studies will be considered in greater detail in the following discussion. In some instances, the dosages studied are in excess of that which might be used for food preservation (approximately four-and-one-half to six megareps); however, these data have been included since they are useful in drawing comparisons and estimating the degree of change that might be expected in the package.

Polystyrene. Polystyrene is of interest because of its remarkable resistance to radiation-induced change. During irradiation in the absence of air (oxygen) *(13, 18)*, the polyethylene molecule undergoes cross linking with the evolution of hydrogen gas. In the presence of air (oxygen), some surface oxidation and chain scission is observed. The stability of polystyrene to radiation-induced change is illustrated by the total energy, 3,000 to 5,000 electron volts, required to produce one crosslink, as compared to the 22 electron volts required to produce one crosslink in polyethylene *(11)*. One explanation for the stability of polystyrene is the presence of the benzene ring as a side chain. The resonance energy of the benzene ring (39 kilocalories per gram mole) is known to stabilize this type of compound *(34, 40)*. When we consider that each polystyrene molecule contains approximately 3,000 benzene rings, it is apparent that this added stability assumes large proportions.

Polyethylene. During irradiation the first noticeable change in the composition of polyethylene is

Table 1. Relative Stability of Various Chemical Groupings (4, 6, 43)

Chemical grouping	Representative type polymer	Dose required to cause 50% loss of tensile strength 10^6 rad	Predominant effect
$-CH(C_6H_5)CH_2-$	Polystyrene	>30	Crosslinking
$-N(C_6H_5)CH_2-$	Aniline formaldehyde resins		
$-CH=CH-$	Present in elastomers (rubbers)		Crosslinking
$-CH_2-CH_2-$	Polyethylene	>10	Crosslinking
$-CO-NH-$	Polyamids such as nylon	>10	
$-Si(CH_3)_2O-$	Methyl silicone		
$-C_6H_3(OH)CH_2-$	Pheno-formaldehyde resin		
$-CH_2O-$	Polyvinyl butyral	0.6	Crosslinking
$-CH_2-S(S)-$	Thiokol		Scission
$-C_6H_5C(O)O-$	Polyesters, dacron	4	Crosslinking
$-CH_2-CHCl-$	Polyvinyl chloride	4	Scission
 Cellulose, sugars, starches			Scission
$-CH_2-CCl_2-$	Saran	1.0	Scission
$-CF_2-CFCl-$	Fluorothane		Scission
$-CF_2-CF_2-$	Teflon	0.07	Scission
$-CH_2-CR_2-$	Polymethyl methacrylate	0.05	Scission

the disappearance of vinylidene end groups *(16)* accompanied by the formation of vinylene groups. Simultaneously, gases are released; the production of gases is linear at doses below 16 megarep *(10)*. The gases produced consist of hydrogen (85 percent) and saturated and unsaturated hydrocarbons with chain lengths of two, three, and four carbon atoms. These short chain hydrocarbons are probably produced from the side chains normally attached to the polyethylene molecule. Of the hydrogen gas that is produced, 70 to 80 percent results from the formation of unsaturated bonds, while the remaining 20 to 30 percent is formed during crosslinking processes. Infrared absorption studies *(5)* have demonstrated that polyethylene is oxidized when irradiated in the presence of oxygen and that oxidation is predominantly on the surface of the material.

The effect of high energy radiation on the functional characteristics of polyethylene, which may influence its effectiveness as a package, is shown in table 2.

As can be seen, the dosage utilized in food processing (approximately six megarep or less), has very little effect on the tensile strength, ultimate elongation, greaseproofness, moisture vapor transmission rate, oxygen permeability, and heat sealability.

The principal difficulty of polyethylene packages lies in the production of off-odors and flavors resulting from gaseous products formed during irradiation. The gaseous products formed will be in-

Table 2. Effect of High Energy Radiation on the Functional Properties of Polyethylene (5, 35, 44)

Dose megarep	Tensile strength lb./sq. in.	Ultimate elongation percent	Grease-proofness seconds	Moisture vapor transmission rate[1]	Sealability after irradiation tensile strength of seal lb./in. width	Oxygen permeability cc./100 sq. in./24 hr. sample 0.002 inch thick
0	2100	650	>1800	1.24	3.98	317
1.0	2180	635	.....	0.99	3.56	.....
3.0	.....	.....	>1800	1.24	4.83	340
6.0	.....	.....	.....	1.13	3.41	.....
10.0	1729	500	.....	.....	.....	.....

[1] 100° F., 90% R.H. in g./100 sq. in./24 hr./0.001 in thickness.

fluenced by the degree of oxidation of the polyethylene prior to irradiation *(7)*. Off odors have been noted in numerous instances, being characterized in some cases as a paraffin odor and in others as a butyric odor. In addition to the odor, an off-flavor has been detected in citrus foods and in some baked items irradiated in polyethylene bags. Furthermore, off-flavors can be detected when water is heated in bags made from some lots of polyethylene. Some of the odors and flavors may not arise from the polymer but may derive from materials added to polyethylene such as antioxidants. With "fresh" stock of known history, off-odors and flavors have not been observed.

Mylar (polyethylene terephthalate). When irradiated in a high neutron flux, Mylar does not evolve appreciable quantities of gas. X-ray diffraction studies have shown that after irradiation Mylar exhibited a well-oriented crystal structure, thereby indicating that crosslinking did not occur *(31)*. It has been demonstrated that the change in Mylar is a scission phenomenon with variable end products depending upon the oxygen tension during irradiation. Irradiation in the absence of air results in an increase in the quantity of carboxyl groups formed; in the presence of air, hydroxyl and carbonyl groups are produced.

Since Mylar is difficult to seal, studies have been conducted utilizing polyethylene-coated Mylar — the polyethylene supplying the means of heat seal. The effects of irradiation on this combination are shown in table 3. The radiation does have a minor effect on the polyethylene

Table 3. Effect of Irradiation on the Functional Properties of 0.0005 Inch Thick Mylar Coated with 0.002 Inch Thick Polyethylene (44, 45)

Dose megarep	Tensile strength lb./in. width	Ultimate elongation percent	Moisture vapor transmission rate[1]	Strength of irradiated seals lb./in. width	Sealability after irradiation strength of seals lb./in. width
0	13.65	87.41	0.53	7.76	7.33
0.2	13.73	84.61	0.53	7.81	8.27
0.7	13.47	89.25	0.51	7.26	7.00
1.3	13.17	81.93	0.50	7.73	8.58
3.3	13.77	81.45	0.50	6.95	5.12
6.5	13.27	82.55	0.56	6.43	5.23

[1] 100° F. 90% R.H. g./100 sq. in./24 hrs.

component; however, this effect does not influence the serviceability of the package.

When polyethylene-coated Mylar packages containing pork sausage or other fatty foods are irradiated, the effects are considerably different. The seals are weakened and delamination is observed, the degree of failure being proportional to irradiation dosage and subsequent storage temperature. Similar results have been encountered with polyethylene-coated cellophanes.

Pliofilm. Irradiation (at three and one-half megarep) of 140-gauge N-2 pliofilm does not appreciably alter the moisture vapor transmission rate *(35)*. When type NO film is irradiated, there is a decrease in tensile strength and elongation accompanied by an increase in sealing temperature.

Halogenated plastics (saran, polyvinylchloride, teflon, fluorothene). Plastics which contain halogens are sensitive to radiation, liberating halogen gases and halogen acids as a result of the radiation. The rate of evolution of fluorine from fluorothene has been determined to be seven millimoles per gram of material per billion roentgens *(10)*. The evolution of gas when teflon is irradiated has been found to conform to the following equation:

$$X = (3.78 \times 10^{-7})\ Y^{1.15}$$

where X is the micrograms of fluorine liberated per gram of teflon and Y the radiation dose in roentgens. In the case of polyvinylchloride the liberation of halogen is measurable at doses as low as one-half megarep *(21)*.

Results have been variable with saran film. In some samples there has been no evidence of deterioration; in others, there has been a marked discoloration at two and one-half megarep. It is probable that formulation, age, and previous history of the film may have an influence on results obtained during irradiation.

Formation of halogen acids in packages made from halogenated films is quite serious since the halogen acid may act as a catalyst in promoting degradation of the package during storage *(17)*.

Table 4 summarizes data in respect to saran film packages.

Table 4. Effect of High Energy Radiation on the Functional Properties of Saran (35)

Dose megarep	Moisture vapor transmission rate[1]	Grease-proofness seconds	Tearing resistance grams
0	0.06	>1800	66
3.5	0.06	>1800	50

[1] 100° F. 90% R.H. g./100 sq. in./24 hr.

Cellulose. Cellulose materials occupy a prominent position among packaging materials. This group consists of papers, waxed papers, greaseproof papers, coated papers, cellophanes, and carton and shipping container stocks. In addition, cellulosic materials are combined with plastics and metal foils for the purpose of providing strength and physical protection to the other components of the combination.

When cellulose materials are subjected to high energy radiation, there is a scission of the molecule. At very high doses, the cellulose is largely converted into water-soluble substances. The decomposition of cellulose has been studied by measuring the increased amounts of sub-

Table 5. Decomposition of Celluloses by Irradiation (38, 39)

Cellulose material	Dose megarep	Decomposition of Carbohydrate percent	Over-all sugar yield percent
Cotton linters	0	....	23.0
	10	2	30.4
Wood Pulp	0	....	33.5
	10	5	44.5
Wood	0	....	25.0
	10	3	36.5
Glucose	10	2	.....

stances which are readily hydrolyzed to form sugars *(28, 38, 39)*. These results are shown in table 5.

Degradation of the cellulose is readily apparent in the loss of tensile strength of papers and cellophanes, as shown in table 6, and by the loss of internal tearing resistance as shown in table 7.

As can be expected, other properties which depend on fiber strength are also degraded; for example, the strength of seals (peel) of polyethylene-coated kraft paper depends on the energy required to separate the fibers. The loss of seal strength is shown in table 8.

Properties which do not depend upon fiber strength are not too seriously affected by radiation. For example, the effect of radiation on greaseproofness is shown in table 9.

The effect of irradiation on the moisture vapor transmission characteristics of cellophane is shown in table 10. This table demonstrates the differences that may be encountered when a material is irradiated by itself as opposed to irradiating it with contents (water, in this case).

Paraffin. Paraffin is used in conjunction with cellulosics to provide a moisture barrier, to provide a means of sealing and, in some cases, as a laminating agent.

In general, the paraffins have a structure similar to that of polyethylene, but differ in molecular weight, degree of branching, and presence of unsaturation. Therefore, it is to be expected that paraffin should react in a manner similar to polyethylene when irradiated. Investigations have demonstrated that paraffins crosslink, forming insoluble gels when irradiated *(13, 14, 41)*.

Laminated Materials. In packaging foods, wide use is made of laminated materials. Usually the laminates consist of a plastic film on the inside, a layer of thin aluminum foil, and a layer of paper, acetate, or scrim cloth on the outside. The purpose of the plastic film is to provide a means of heat sealing and chemical protection to the aluminum foil; the aluminum foil in turn provides the required moisture or oxygen resistance, and the paper, acetate, or cloth provides mechanical strength. The effect that high energy radiations would have on laminations depends upon the individual components and the adhesives used in bonding the plies. However, the effect of high energy radiation on

Table 6. Tensile Strength of Irradiated Cellulosic Material (35, 47) (pounds/inch of width)

Dose megarep	35 pound kraft coated with 18 pounds polyethylene	450 gauge MSAT cellophane	MSB-3 cellophane	Bleached glassine
0	48.38	24.4	24.8	13.4
2	44.65	.....	.....	.....
3.5	.....	16.0	21.8	14.5
5	40.86	.....	.....	.....
10	39.61	.....	.....	.....

Table 7. Internal Tearing Resistance of Irradiated Cellulosic Materials (35) (grams)

Dose megarep	35 pound kraft coated with 18 pounds polyethylene	300 gauge MSAT-83 cellophane	450 gauge MSAT-80 cellophane	300 gauge MSAT-54 cellophane
0	93	11	14	17
3.0	84	8	6	6

Table 8. Tensile Strength (Peel) of Irradiated Seals (47) (35 pounds per ream kraft paper coated with 18 pounds per ream polyethylene)

Dose megarep	Tensile strength lb./in. width
0	3.71
2	3.36
5	3.26
10	2.81

Table 9. Effect of Radiation on the Greaseproofness of Cellulose Materials (35) (greaseproofness in seconds)

Cellulose material	Dose 0 rep	Dose 3 megarep
Waxed paper (26 pound sulphite—9 pound paraffin wax)	340	320
35 pound kraft coated with 18 pounds polyethylene	>1800	>1800
Bleached glassine	>1800	>1800
P-1 Cellophane	>1800	>1800
MSB-3 Cellophane	>1800	>1800

Table 10. Moisture Vapor Transmission Rate of Irradiated Cellophane (22) (g./100 sq. in./24 hrs. 100°F. — 90% R.H.)

Grade cellophane	Contents of package	Dose megarep 0	3.5	7.0
MSAT-87	none	1.1	1.4	2.4
300 gauge	water	2.5	3.2	7.8
MSAT-80	none	1.7	....	3.8
300 gauge	water	2.9	4.5	8.0

Table 11. Loss of Adhesion of Vinyl Component During Irradiation

Dose megarep	0.0015-inch thick vinyl laminated to 0.001-inch aluminum laminated to 44 x 36 scrim peel strength vinyl component lbs./in. width
0	0.84
10	0.028

Prior to joining the QMFCI staff in 1949 Mr. George E. Tripp had a varied background which included research on amino acids and rates of aminolysis, waterproofing of leather and textile goods, emulsion polymerization, and the relationships existing between packages and packaged food. Since joining the Institute staff he has been engaged in research related to food packaging. Mr. Tripp has authored numerous technical papers on packaging, containers and related studies. A native of Chicago, he was graduated from the University of Illinois in 1939 with a B.S. in chemistry. Prior to World War II Mr. Tripp was employed by the Ordnance Corps. Shortly after the outbreak of hostilities he enlisted in the Army Air Force and received his commission as a meteorologist. Mr. Tripp is a packaging technologist, Container Laboratories, QMFCI.

Mr. John P. Crowley has been with the Container Laboratories, QMFCI, since May 1956. As a native of Chicago, he was graduated from the University of Illinois in June 1954 with a B.S. in chemistry and zoology. He received his M.S. in microbiology and genetics from the University of Illinois in February 1956. Mr. Crowley served with the U. S. Army Medical Corps from October 1950 to November 1952, serving for one year in Korea and Japan. Prior to entering the Army he was employed as a laboratory technician with the U. S. Rubber Co., Developmental Laboratory in Chicago.

adhesives needs further exploration. An example of the effect on one lamination is shown in table 11.

impact on packaging

The use of high energy radiation in preserving foods will have a profound influence on food packaging. New designs of containers will make their appearance, and flexible containers will find new applications.

Packages designed for irradiated foods will have to take into consideration the nature of radiation and its attenuation in the food.

When processing food with high energy radiation, we find an analogy to heat processing; namely, the presence of "hot" and "cold" spots in the container. The shape and dimensions of the container will be determined by the dose range that the food can tolerate; the minimum dose being that required for sterilization, and the maximum being the dose which will result in the food becoming unacceptable. Current objectives are to maintain the dose variation between minus zero and plus ten percent, with plus 25 percent being the maximum acceptable variation.

The shape of the container will be determined by the configuration of the source. As beta generations scan the package with a beam of electrons, the ideally shaped container, in this instance, would have a rectangular cross section. Width of the container would be governed by the scanning width of the beam, and the depth determined by the energy of the electron beam and the angle of incidence at the outermost limits of the beam. In the case of gamma radiation, one possible source would be an indium sulfate solution activated by neutrons. If the container were to be passed directly through the solution, the best shape would be a sphere. However, if the radioactive solution were to be circulated around a tube and the containers passed through the tube, the ideal container shape would then be a cylinder. Over-all dimensions of the container would be determined by the attenuation of the radiation in the container and contents, and the maximum dose variation that the food could tolerate.

Dose distribution and efficiency are affected by container construction materials; the absorber densities of some container materials are listed in table 12.

Table 12. Absorber Densities of Some Package Materials

	Package	Absorber Density g./sq. cm.
Papers:		
18-lb. per ream kraft or sulphite		0.003
26-lb. per ream kraft or sulphite		0.0035
26-lb. per ream kraft or sulphite coated with 9-lb. per ream paraffin		0.005
40-lb. per ream kraft or sulphite		0.006
60-lb. per ream kraft or sulphite		0.009
Bending Boards:		
33-lb. per 1000 sq. ft.		0.016
65-lb. per 1000 sq. ft.		0.032
90-lb. per 1000 sq. ft.		0.044
Plastic Films:		
Polyethylene	0.001-inch thick	0.0023
Saran	0.001-inch thick	0.0044
Polyvinyl chloride	0.001-inch thick	0.0035
Pliofilm	0.001-inch thick	0.0028
Mylar	0.001-inch thick	0.0035
Cellophane	300-gauge MST-53	0.0035
Cellophane	450-gauge MSAT-80	0.0049
Metals:		
Aluminum	0.012-inch thick	0.0824
Steel	60-gauge (0.0066-in. thick)	0.134

Additionally, the use of ionizing radiation will introduce new uses for flexible containers. Previously, flexible containers have not been used for sterilized foods since they did not have the necessary heat resistance or physical characteristics to withstand conventional retorting. Radiation processing removes the need of the container to withstand the heat and stress encountered in conventional processing. Radiation will also enable sterilization of the

container itself by means other than heat *(23, 24)*. This would make possible the aseptic packaging of foods in plastic films, the foods being first processed either by radiation or by means of conventional aseptic canning systems now in use.

problems and some probable answers

A considerable amount of information has been accumulated; however, there are several important problems which require resolution. These are:

1. the possibility that the food can interact with the package during or after irradiation to the detriment of food and package (data thus far accumulated are based on the irradiation of packaging out of contact with the food);
2. the ability of bacteria to penetrate thin plastic film packages used for sterile foods is still undetermined;
3. a positive method of sealing flexible and semi-rigid plastic containers, particularly when food particles are present in the area to be sealed, is another unanswered need;
4. new packaging and design concepts to take advantage of the benefits of radiation processing;
5. minimum respiration rate required to prevent undesirable changes in the food while increasing its shelf life (this area requires extensive investigations for conventional as well as irradiated foods);
6. some insect species are capable of penetrating thin plastic containers. Increased use of flexible containers for food will intensify this problem.

With respect to current packaging for irradiated foods, experience and information is limited. However, based on the knowledge available, existing commercial packaging probably could serve as shown for the following types of irradiated foods:

Tubers treated for sprout inhibition. Normal commercial crates as well as burlap bags and multiwall paper sacks probably will be suitable. The latter should not contain an asphalt laminated ply as it would hold moisture within the sack and create a condition favorable for mold growth and rot.

Grains treated for insect control. The grain could be irradiated in bulk and packaged (after irradiation) in common commercial packages. To prevent reinfestation, the package should be insect-proof or treated with insect-resistant materials. Where the grain is irradiated in the package, the dimensions of the package will have to be adjusted to the type and range of the radiation. Containers such as metal cans, insect-resistant-treated, multiwall paper shipping sacks, and paper pockets and cartons, overpacked in insect-resistant-treated multiwall bags or shipping containers, should all prove suitable.

Pasteurized foods (Doses of 1 megarep). Current commercial packages should prove adequate. Principal problems are (a) the possible interaction of the package and food resulting in food off-flavors, odors, or color deterioration, and the further possibility of the interaction damaging the package, (b) the ability of the package to prevent bacterial contamination of the food, and (c) modifications of the type of package required for the food through the changed chemistry of the food.

Sterilized foods. (Doses of 4.5 to 6 megarep). Less is known in this area. Currently, polyethylene-coated aluminum laminated to Mylar film should be satisfactory as a flexible package for some foods, and commercial type, tin-plated cans should be satisfactory as rigid containers. The principal problems in this area are (a) positive seals, adequate resistance to damage, prevention of bacterial contamination of flexible containers, and (b) in the case of thermal processed canned foods experience has shown that there is an interdependence of the type of steel,

tin coating weight, and types of enamels (if used) and the food; this relationship must be investigated in respect to foods processed by radiation to determine if these factors will differ with the irradiated food.

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